

**Analytical Chemistry Information Summary and Proposed Compounds for Monitoring COREXIT for Deepwater Horizon
Gulf of Mexico Oil Spill – Interim Final, June 2010**

Table 1. Summary of available analytical method information for COREXIT compounds.

	CAS	Name(s)	Analysis method	Detection/ Reporting Limit	Comments
1	57-55-6	Propylene glycol; 1,2-propanediol	EPA R6: GCMS, direct injection, wax column	DL = 0.5 ppm	EPA R6: Robust method for water and sediment samples (water extract of sediment) Very commonly used compound, questionable for use as a marker. But useful in weight of evidence for COREXIT presence. Same run as for di(propylene glycol) butyl ether and 2-BE. THIS IS THE RECOMMENDED METHOD
			Accutest NJ: GC/MS SIM 8260, direct aqueous injection	DL for Accutest = 80 ppb; RL = 0.5 ppm	RTX200 column
			ALS: GC/MS, direct injection, wax column	DL = 0.5 ppm	
2	111-76-2	2-Butoxyethanol; "2-BE"	EPA R6: GC/MS, direct injection, wax column	DL = 0.5 ppm	EPA R6: Quantitative method for 2-BE from water and sediment. Same run as for di(propylene glycol) butyl ether and propylene glycol.

			EPA R5: LC/MS/MS, direct injection	RL = 125 ppb	Not ready yet for samples (ready by end of June). Same run as for di(propylene glycol) butyl ether. THIS IS THE RECOMMENDED METHOD [may switch to GC/MS method later if final screening values allow higher DL and initial monitoring results support the switch]
			Accutest FL: GC/FID 8015, direct aqueous injection	DL = 0.5 ppm; RL = 2.0 ppm	Accutest: Robust method; DB-Wax column
			ALS: GC/MS, direct injection, wax column	DL = 0.5 ppm	
3	577-11-7	Dioctylsulfosuccinate sodium salt; Bis(2-ethylhexyl) sulfosuccinate sodium salt; Dioctyl sodium sulfosuccinate; Docusate sodium; "DOSS"	EPA R5 and R6: LC/MS/MS, direct injection	DL = 3 ppb; RL = 20 ppb	Ready for use on seawater/oil samples. Extraction method has options for water only, oil only, or total sample (including sample bottle as per NALCO recommendations). THIS IS THE RECOMMENDED METHOD
			Columbia Analytical Services: LC/MS/MS	DL seawater 100 pptrillion	CAS can analyze DOSS in seawater and sediment; working on tissue method.
4	1338-43-8	Sorbitan Monooleate, "SPAN 80"	Nalco: LC/MS with positive ion detection, surfactant column by Dionex,		Little method development reported for this compound in environmental matrices, although there is interest in potentially using it as a COREXIT marker, as it is an individual compound.
			Ed Furlong, USGS:		Ed sees 2 compounds when analyzing this.
5	9005-65-6	Polyoxyethylenesorbitan monooleate; Sorbitan monooleate ethoxylate; "TWEEN 80"	?		No method development reported for this compound. This is a mixture of compounds, and difficult to analyze.

6	9005-70-3	Polyethylene glycol sorbitan trioleate; Polyoxyethylene sorbitan trioleate; Polysorbate 85; "TWEEN 85"	?		No method development reported for this compound. This is a mixture of compounds, and difficult to analyze.
7	29911-28-2	2-Propanol,1-(2-butoxy-1-methylethoxy)-; Di(Propylene Glycol) Butyl Ether; "2-P"	EPA R6: GC/MS, direct injection, wax column	DL = 0.5 ppm	EPA R6: Robust method for water and sediment samples (water extract of sediment). Same run as for 2-BE and propylene glycol.
			EPA R5: LC/MS/MS, direct injection	RL = 1 ppb	Not ready yet for samples (end of June). Same run as for 2-BE. THIS IS THE RECOMMENDED METHOD [may switch to GC/MS method later if final screening values allow higher DL and initial monitoring results support the switch]
			ALS: GC/MS, direct injection, wax column	DL = 0.5 ppm	
			Accutest		Standards we have tried are all a "mix of isomers" so we are seeing 2 and 3 peaks.
			Battelle: GC/MS-SIM	100-200 ppb	By SIM they are seeing 2 different compounds.
8	64742-47-8	Hydrotreated light distillate			This will be indistinguishable from oil sources
9	104-76-7	2-Ethylhexanol; 2-Ethylhexan-1-ol; 2-Ethyl-1-hexanol	EPA R6: 8260 Purge and Trap GC/MS	EPA R6 water DL= 10 ppb	EPA R6: Heated purge to 80° C is key for method. R6 method in development, likely ready for use soon on water (and other?) samples. THIS IS THE RECOMMENDED METHOD
			EPA R5: GC/MS	RL = 10 ppb	Vacuum distillation concentrator (relatively unusual piece of equipment). Not ready yet for samples (ready by end of June).
			Accutest FL: GC/FID 8015, direct aqueous injection	DL for Accutest = 0.5 ppm; RL = 2.0 ppm	Accutest: Robust method; DB-Wax column.
			ALS: GC/MS	ALS: water	

				RL = 830 ppm	
10	115960-17-3?	Monooctyl sulfosuccinate; "MOSS"?	NALCO: LC/MS	None developed yet	A primary degradation (hydrolysis) product of DOSS. Recommended by NALCO as a potential marker for DOSS in water. Successful development of an analysis method likely if pursued. No method currently available, and may require its own separate analysis.

Table 2. Monitoring plan analyte recommendations:

	CAS	Name(s)	Comments/Recommendation
1	57-55-6	Propylene glycol; 1,2-propanediol	EPA has robust method, but DL is high. Very widely used compound, low toxicity. Low utility as a COREXIT marker, only analyze as part of “2-BE” and “2-P” analysis. RECOMMENDATION: Analyze as part of surface water monitoring program.
2	111-76-2	2-Butoxyethanol; “2-BE”	EPA has robust method, but DL is high. Potentially good marker for COREXIT 9527, and fairly toxic compound. COREXIT 9527 reportedly not being used any more – still keep? RECOMMENDATION: Analyze as part of surface water monitoring program.
3	577-11-7	Diocylsulfosuccinate sodium salt; Bis(2-ethylhexyl) sulfosuccinate sodium salt; Dioctyl sodium sulfosuccinate; Docusate sodium; “DOSS”	NALCO, Battelle, Columbia Analytical Services, others feel that this will be the key COREXIT marker compound for analysis, especially in water. LC/MS/MS is the key method. EPA has a robust method for water analysis. RECOMMENDATION: Analyze as part of surface water and sediment monitoring program.
4	1338-43-8	Sorbitan Monooleate, “SPAN 80”	NALCO, others believe this compound has potential utility as a COREXIT marker compound in surface water, as it is a discrete compound. Wide use makes utility as a diagnostic marker low near shore, but ok offshore. Likely would require a special analysis, which increases costs of inclusion. RECOMMENDATION: Do not use as part of the monitoring program.
5	9005-65-6	Polyoxyethylenesorbitan monooleate; Sorbitan monooleate ethoxylate; “TWEEN 80”	Generally believed that the wide use of this compound means that it will not be a diagnostic marker, and the fact it is a mixture makes analysis from environmental samples very difficult. Compounds also do not appear to be appreciably toxic to aquatic organisms. RECOMMENDATION: Do not use as part of the monitoring program.
6	9005-70-3	Polyethylene glycol sorbitan trioleate; Polyoxyethylene sorbitan trioleate; Polysorbate 85; “TWEEN 85”	Generally believed that the wide use of this compound means that it will not be a diagnostic marker, and the fact it is a mixture makes analysis from environmental samples very difficult. Compounds also do not appear to be appreciably toxic to aquatic organisms. RECOMMENDATION: Do not use as part of the monitoring program.
7	29911-28-2	2-Propanol,1-(2-butoxy-1-methylethoxy)-; Di(Propylene Glycol)	NALCO, others believe this is a decent marker for COREXIT. EPA has a robust method for water and sediment, although the detection limits are

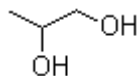
		Butyl Ether; "2-P"	high. Battelle has measured this in water near area of dispersant application. RECOMMENDATION: Analyze as part of surface water and sediment monitoring program.
8	64742-47-8	Hydrotreated light distillate	RECOMMENDATION: No utility in monitoring program due to similarity with oil.
9	104-76-7	2-Ethylhexanol; 2-Ethylhexan-1-ol; 2-Ethyl-1-hexanol	Likely environmental degradation product of COREXIT (DOSS, specifically) in surface water, therefore likely a good marker compound. EPA is developing a method for water analysis. RECOMMENDATION: Analyze as part of surface water (and sediment?) monitoring program.
10	115960-17-3?	Monooctyl sulfosuccinate; "MOSS"?	Probable primary degradation (hydrolysis) product of DOSS in surface water. NALCO could help to develop environmental analytical method, but no method currently exists. RECOMMENDATION: Do not use as part of the monitoring program at this time. [Potentially consider later if methods become available.]

* Fumarate/maleate from GC analysis of DOSS initially considered as a DOSS indicator. NALCO indicated that this is a degradation (desulfonation) of DOSS due to high injector/detector temps. They also indicated that the desulfonation was not consistent, making the quantitation using the fumarate/maleate problematic. The fumarate/maleate are therefore not proposed for inclusion into the monitoring program at this time.

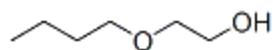
Table 3. SUMMARY OF COMPOUNDS RECOMMENDED FOR DISPERSANT MONITORING:

No.	CAS	Name(s)	Recommended Method	Detection/Reporting Limit	Comments
1	57-55-6	Propylene glycol; 1,2-propanediol	EPA R6: GCMS, direct injection, wax column	DL = 0.5 ppm	Robust method for water and sediment samples (water extract of sediment)
2	111-76-2	2-Butoxyethanol; "2-BE"	EPA R5: LC/MS/MS, direct injection	RL = 10 ppb	Not ready yet for samples (ready by end of June). May switch to GCMS method (cheaper, faster than LC/MS/MS?) if final screening values allow higher DL and initial monitoring results support the switch.
3	577-11-7	Dioctylsulfosuccinate sodium salt; Bis(2-ethylhexyl) sulfosuccinate sodium salt; Dioctyl sodium sulfosuccinate; Docusate sodium; "DOSS"	EPA R5 & R6: LC/MS/MS, direct injection	DL = 3 ppb; RL = 20 ppb	Ready for use on seawater/oil samples.
7	29911-28-2	2-Propanol,1-(2-butoxy-1-methylethoxy)-; Di(Propylene Glycol) Butyl Ether; "2-P"	EPA R5: LC/MS/MS, direct injection	RL = 5 ppb	Not ready yet for samples (ready end of June). May switch to GCMS method (cheaper, faster than LC/MS/MS?) if final screening values allow higher DL and initial monitoring results support the switch.
9	104-76-7	2-Ethylhexanol; 2-Ethylhexan-1-ol; 2-Ethyl-1-hexanol	EPA R6: 8260 Purge and Trap GC/MS	water DL= 10 ppb	EPA R6: Heated purge to 80° C is key for method. R6 method in development, likely ready for use soon on water (and other?) samples.

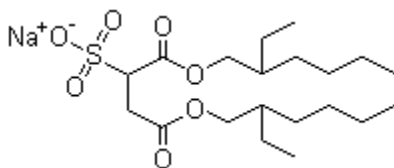
Appendix 1. Structures for reference:



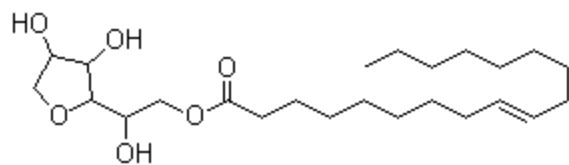
1) Propylene glycol; 1,2-propanediol; 57-55-6 ^A



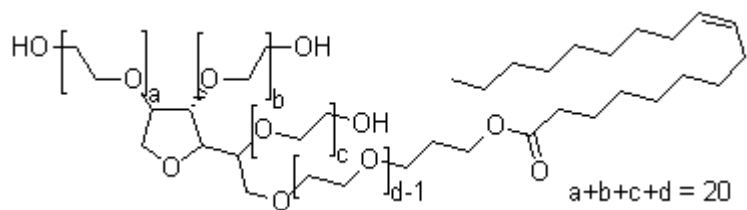
2) 2-Butoxyethanol; 111-76-2 ^B



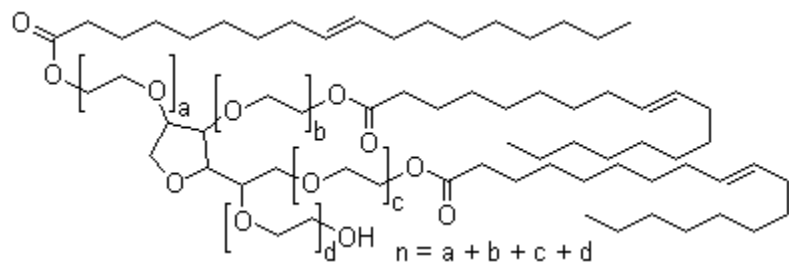
3) Dioctylsulfosuccinate sodium salt; Bis(2-ethylhexyl) sulfosuccinate sodium salt; Dioctyl sodium sulfosuccinate; "DOSS"; 577-11-7 ^A



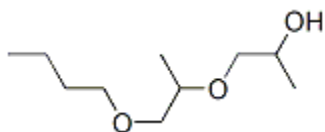
4) Sorbitan Monooleate; 1338-43-8 ^A



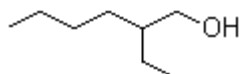
5) Sorbitan monooleate ethoxylate; TWEEN 80; 9005-65-6 ^A



6) Polyethylene glycol sorbitan trioleate; Polyoxyethylene sorbitan trioleate; Polysorbate 85; TWEEN 85; 9005-70-3 ^A



7) 2-Propanol,1-(2-butoxy-1-methylethoxy)-; Di(Propylene Glycol) Butyl Ether; 29911-28-2 ^B



9) 2-Ethylhexanol; 2-Ethylhexan-1-ol; 2-Ethyl-1-hexanol ^A

^A Chemical structure figure copied from www.chemblink.com

^B Chemical structure figure copied from www.chemicalbook.com